UNITED STATES

DEPARTMENT OF THE INTERIOR

BUREAU OF MINES

NASA Contract R-09-040-001

MULTIDISCIPLINARY RESEARCH LEADING TO
UTILIZATION OF EXTRATERRESTRIAL RESOURCES

Quarterly Status Report
July 1, 1968 to October 1, 1968

TWIN CITIES MINING RESEARCH CENTER

Walter E. Lewis, Research Director

NASA Contract R-09-040-0Q1   C (

MULTIDISCIPLINARY RESEARCH LEADING TO
UTILIZATION OF EXTRATERRESTRIAL RESOURCES

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U.S. Bureau of Mines NASA Program of Multidisciplinary Research
Leading to Utilization of Extraterrestrial Resources

QUARTERLY STATUS REPORT   July 1, 1968 to October 1, 1968

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STATUS REPORT FIRST QUARTER FISCAL YEAR 1969

U. S. Bureau of Mines NASA Program of Multidisciplinary Research
Leading to Utilization of Extraterrestrial Resources

October 1, 1968

Task title: Background analysis and coordination
Investigator: Thomas C. Atchison, Program Manager
Location: Twin Cities Mining Research Center
                 Minneapolis, Minnesota
Date begun: April 1965   To be completed: Continuing

Personnel: Thomas C. Atchison, Supervisory Research Physicist
                 Other Bureau personnel, as assigned

PROGRESS REPORT

Objective

The objective of the program is to help provide basic scientific and
engineering knowledge needed to use extraterrestrial mineral resources
in support of future space missions. Under this task, background and
supporting studies and coordinating and liaison activities for the
program are carried out.

Progress During the First Quarter

Background and supporting studies included in this task were formerly
reported under three tasks covering core group activities, selection
and collection of simulated lunar materials, and physical properties
of simulated lunar materials in earth environment. Actual property
measurement work is being reported under the most closely related re-
search task.

A summary of the evaluations made by the NASA panel that reviewed the
Bureau's program last May was received from NASA Headquarters in Au-
gust. The summary complimented the Bureau researchers on an excellent
presentation of their work. It also contained some helpful suggestions.
In general the panel recommended emphasis on the more basic studies of
rock properties and behavior in simulated lunar environment. A propos-
al for continuing the Bureau's program into fiscal year 1970 and 1971,
after completion of the current research tasks, was prepared and submit-
ted to NASA in July.

Plans were made to replenish the supply of our 14 simulated lunar rocks
being used in the Bureau's program and by other groups doing related re-
search. D. E. Fogelson and B. L. Vickers, with the cooperation of the

Bureau's Spokane Mining Research Laboratory and Albany Metallurgy Research Center, plan a trip through Oregon and California beginning September 30 to collect several tons of needed rocks.

Word was received from Dr. Jane de Wys of the Jet Propulsion Laboratory, Pasadena, Calif., on progress in the Surveyor simulation studies on the 14 simulated lunar rocks. Simulated footpad magnet experiments have been completed on fine particle samples of the 14 rocks, and the gabbro, flow basalt, and vesicular basalt #1 most closely duplicate the Surveyor V, VI, and VII lunar landing results.

Status of Manuscripts

Proposal for Continuing Bureau Extraterrestrial Resource Utilization Program, by T. C. Atchison, was submitted to NASA in July.

STATUS REPORT FIRST QUARTER FISCAL YEAR 1969

Bureau of Mines NASA Program of Multidisciplinary Research

Task title: Chemical reactivity of freshly formed surfaces
Investigator: Clifford W. Schultz, Project Leader
Location: Twin Cities Mining Research Center
                Minneapolis, Minnesota
Date begun: April 1966   To be completed: June 1969

Personnel: Clifford W. Schultz, Metallurgist
                William H. Engelmann, Research Chemist

PROGRESS REPORT

Objective

The objective of this task is to define the equilibrium state of mineral surfaces with respect to adsorbed gases. Further, it is intended that existing adsorption isotherms be clarified in the low pressure end of the scale.

Progress During the First Quarter

This project has been recessed during the first quarter. Work planned for the first and second quarters is now planned for accomplishment during the second quarter.

Clifford W. Schultz, the original investigator for this and the following task, has been assigned to full-time research duties and will be completing requirements for his doctor's degree. William H. Engelmann has assumed supervision of the chemical fragmentation and surface property research at the Center. Mr. Schultz will continue as leading investigator for this task.

Status of Manuscripts

None scheduled until the fourth quarter.

STATUS REPORT FIRST QUARTER FISCAL YEAR 1969

Bureau of Mines NASA Program of Multidisciplinary Research

Task title: Surface properties of rock in lunar environment
Investigator: Wallace W. Roepke, Project Leader
Location: Twin Cities Mining Research Center
                     Minneapolis, Minnesota
Date begun: April 1966   To be completed: July 1969

Personnel: William H. Engelmann, Research Chemist
                     Wallace W. Roepke, Principal Vacuum Specialist
                     Kenneth G. Pung, Physical Science Technician

PROGRESS REPORT

Objective

The goal of this task is to measure the surface properties of rocks and
rock-forming minerals in a simulated lunar environment. Because fric-
tion is an important feature of many mining operations, the immediate
objective is to measure the coefficients of friction of several mineral
and mineral-metal (tungsten carbide) pairs in ultrahigh vacuum.

Progress During the First Quarter

Three primary sensors for the friction test apparatus have been designed
which will give improved sensitivity. Two have been fabricated and are
being calibrated. The output of the heavy load bridge is 38 g/mv. The
medium load bridge has not yet been fully calibrated and the light load
bridge is still being assembled.

The friction test apparatus, minus the driving mechanism on the rotary
feed-throughs, has been placed in the ultrahigh vacuum chamber to obtain
preliminary pumpdown data. The baked system responded with the same
speed to system pumpdown as the normal empty chamber and the ultimate
pressure was the same. Some minor modifications on the driving mechanism
for the friction test apparatus have required additional work in the ma-
chine shop. These involve coupling adaptors which allow the apparatus to
be repositioned in the chamber. This will permit use of a possible laser
cleaning technique without complicated internal optics to position the
beam.

A Coherent Radiation Laboratories laser which has been purchased by the
Bureau is being evaluated for use as a primary cleaning technique for
the ultrahigh vacuum friction work. Initial studies for generating clean
surfaces in ultrahigh vacuum have been started. The variable parameters
are pulse duration, pulse current, and beam diameter (focused or unfo-
cused). Incident light and polarized light microscopy of surfaces prior

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to irradiation has been initiated. Photographic records of these surfaces are being made before and after the laser bombardment. Front surface temperature is being measured initially by optical pyrometry while heat transfer through the sample is measured at the back surface with small thermocouples.

Cholesteric liquid crystals are also being used for back surface temperature measurement. Liquid crystals show changes in color for small changes in temperature. This nondestructive measurement method has several advantages over thermocouples. Chief among these is the fact that the liquid crystal material is brushed onto the surface, providing intimate contact. Avoiding heat losses from thermocouple leads and variations caused by uneven contact area are further advantages. The response is fast and reversible so the sample may be cycled an infinite number of times.

Infrared windows for the ultrahigh vacuum system are in the design stage. These will probably be of Irtran 4 (Kodak trade name) mounted in Pyrex to allow visual and infrared use simultaneously. Preliminary testing has begun to verify the design.

Assistance was given to the lunar drilling task in planning for drilling experiments in ultrahigh vacuum and to the rock failure task in evaluating scanning electron microscope equipment.

Mr. Roepke has taken over from Mr. Schultz the role of leading investigator for this task.

Status of Manuscripts

Inexpensive Oil Vapor Trap for Use with Rotary Vacuum Pumps, by W. W. Roepke and K. G. Pung, was published in the International Journal of Vacuum in August.

Mass Spectrometer Studies of Outgassing from Simulated Lunar Materials in Ultrahigh Vacuum, by W. W. Roepke and C. W. Schultz, is being prepared for publication in the Journal of the American Vacuum Society.

Vacuum Technology Course, an internal report by W. W. Roepke, is in preparation.

STATUS REPORT FIRST QUARTER FISCAL YEAR 1969

Bureau of Mines NASA Program of Multidisciplinary Research

Task title: Rock failure processes and strength and elastic
                properties in lunar environment
Investigator: Egons R. Podnieks, Project Leader
Location: Twin Cities Mining Research Center
                Minneapolis, Minnesota
Date begun: June 1966   To be completed: June 1969

Personnel: Egons R. Podnieks, Mechanical Research Engineer
                Robert J. Willard, Geologist
                Richard E. Thill, Geophysicist
                Peter G. Chamberlain, Geophysicist
                Rollie C. Rosenquist, Engineering Technician

PROGRESS REPORT

Objective

The objective of this project is to study the effect of ultrahigh vac-
uum on rock deformation and failure processes at the macrostructural
and the microstructural level and to measure rock strength and elastic
properties in ultrahigh vacuum environment.

Progress During the First Quarter

A recently received alphatron gage is being incorporated in the ultra-
high vacuum system to continuously record vacuum over a wide range of
pressure variations. The gage will permit vacuum to be recorded even
during heavy outgassing of the rock core. The outgassing occurs under
increasing load and is especially prominent near failure.

Additional pumpdown curves have been obtained to determine the effect
of preconditioning on pumpdown characteristics and base pressure.

A newly developed technique for simultaneously measuring shear and lon-
gitudinal pulse transit times in rock will be used to obtain concurrent
measurements of dynamic and static mechanical properties of rock under-
going deformation in the vacuum chamber.

R. J. Willard conducted transgranular-intergranular (T/I) and defect
frequency orientation (DFO) analyses on dacite disks previously point
loaded to failure under a medium vacuum. Results of the T/I analysis
indicate that in the vacuum environment fractures tend to traverse
pores rather than the glassy matrix, presumably because moisture re-
moval strengthens the matrix. The DFO analysis revealed a marked fab-
ric anisotropy which appeared to influence fracture direction in dacite.

Status of Manuscripts

Environmental Effects on Rock Properties, by E. R. Podnieks, P. G. Chamberlain, and R. E. Thill, has been submitted for publication in the Proceedings of the Tenth Symposium on Rock Mechanics.

STATUS REPORT FIRST QUARTER FISCAL YEAR 1969

Bureau of Mines NASA Program of Multidisciplinary Research

Task title: Feasibility of thermal fragmentation studies in vacuum
Investigator: Kuppusamy Thirumalai, Project Leader
Location: Twin Cities Mining Research Center
                Minneapolis, Minnesota
Date begun: October 1966   To be completed: June 1969

Personnel: Joseph M. Pugliese, Geophysicist
                Kuppusamy Thirumalai, Mining Engineer
                Russell E. Griffin, Electronic Research Engineer
                Sam G. Demou, Physicist

PROGRESS REPORT

Objective

The objective of this task is to investigate the feasibility of extending thermal fragmentation studies to lunar vacuum environment.

Progress During the First Quarter

Experiments were continued to determine the thermal softening limits in granodiorite and to confirm the difference in thermal softening limits between nitrogen atmosphere and vacuum conditions observed previously.

Eight 1/2-inch diameter granodiorite cores 5 inches long were drilled with a known orientation out of the same block. The samples were held on zirconium supports with a centrally distributed load of 53 grams suspended by a 1/2-inch molybdenum ribbon around the middle of the specimen. Shock furnace heating was programmed at 5°C/min and similar experimental conditions were maintained for nitrogen atmosphere and vacuum conditions.

Table 1 shows the failure temperatures in nitrogen atmosphere and vacuum environment. The results indicate a general tendency toward higher thermal softening limits in the vacuum environment. Figure 1 shows the deformation at the middle of the specimen during the experiment. The initial positive deformation is attributed to the volume change at increased temperatures. The failure in nitrogen atmosphere appears more gradual than in the vacuum environment. The granodiorite experimental results indicate the probable spalling action for an incident heat flux in vacuum environment is at least as favorable as in atmosphere. Differential thermal analysis for the granodiorite will be carried out during the second quarter to ascertain the reactions at different levels of temperature up to the thermal softening limits.
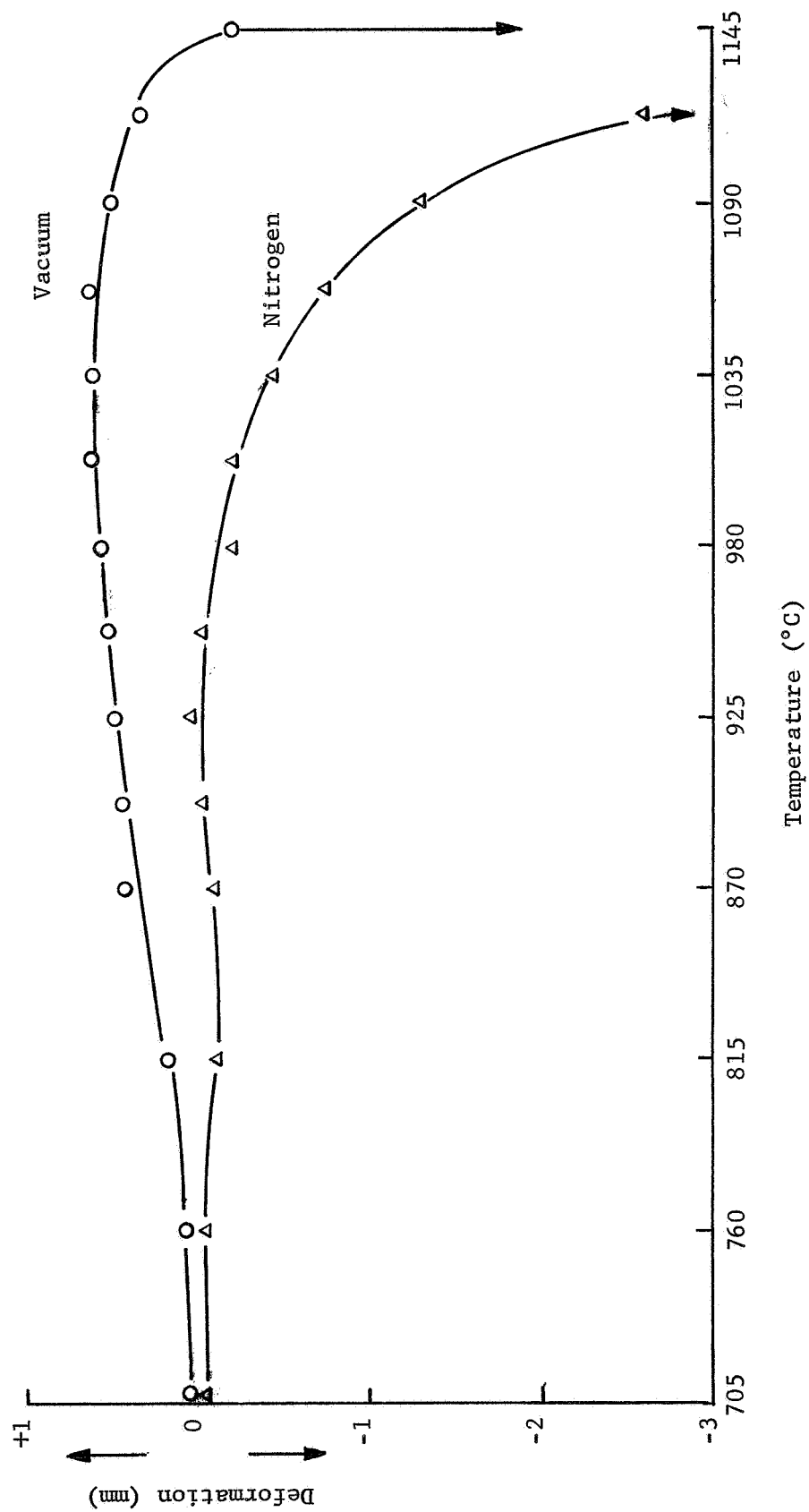
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FIGURE 1. - Deformation Plot for Granodiorite.

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TABLE 1. - Thermal softening of granodiorite

Sample	Environment	Pressure	Temperature °C
		10^{-5} torr	
1	Vacuum	10	1175
2		6	1120
3		9	1160
4		6	1150
		psi	
5	Nitrogen	20	1105
6		20	1135
7		20	1135
8		20	1105

Incident heat energy by laser irradiation provides a convenient form of thermal energy for experiments under vacuum conditions. Investigations of the effect of thermal fragmentation by laser irradiation of simulated lunar rocks at atmospheric conditions have been initiated during this quarter with a view to extending the tests later to vacuum environment. Preliminary results of laser irradiation on tholeiitic basalt, vesicular basalt, dacite, obsidian, and pumice were obtained at energy levels of 50, 100, and 100,000 watts/cm^2 in cooperation with Gulf General Atomic Laboratories. The rock samples were irradiated with a focused as well as partially focused continuous CO_2 laser beam. Fragmentation appeared most prominent at energy levels of 100 watts/cm^2 for all the rocks tested.

A systematic study of rock damage by laser irradiation in Dresser basalt, dacite, and obsidian has begun at our laboratories with a newly acquired 100-watt CO_2 laser. Samples were exposed to continuous irradiation for 10- and 30-second periods and also manually pulsed with pulses of 1-second duration with a constant incident energy of 55 watts/cm^2. A significant increase in damage with pulsed mode was observed in obsidian. A detailed study of the effect of energy level, pulse duration, and pulse shape in these rocks will be conducted during the second quarter.

Status of Manuscripts

None scheduled until fourth quarter.

STATUS REPORT FIRST QUARTER FISCAL YEAR 1969

Bureau of Mines NASA Program of Multidisciplinary Research

Task title: Thermophysical properties of rock over lunar
                temperature range.
Investigator: David P. Lindroth, Project Leader
Location: Twin Cities Mining Research Center
                Minneapolis, Minnesota
Date begun: October 1966   To be completed: June 1969

Personnel: Joseph M. Pugliese, Geophysicist
                Russell E. Griffin, Electronic Research Engineer
                David P. Lindroth, Physicist
                Carl F. Wingquist, Physicist
                Walter G. Krawza, Engineering Technician

PROGRESS REPORT

Objective

The objective of this work is to extend current studies of the effect
of temperature on thermophysical properties of rocks at atmospheric
pressure to cover the lunar temperature range.

Progress During the First Quarter

D. P. Lindroth and W. G. Krawza have completed checkout of the laser
system recently purchased by the Bureau. A flexible sample holder was
constructed to hold specimens up to 2.5 cm in diameter. Initial ther-
mophysical property measurements have been carried out on three disks
of Sioux quartzite using microminiature grid type thermocouples. The
quartzite was chosen because of its homogeneity and because the disks
were readily available. At this point the data show significant scat-
tering. We believe this is due mainly to differences in the thermo-
couples and to the way in which the thermocouples were bonded to the
rear face of the samples. The best way to circumvent these problems
appears to require a good infrared radiometer with a thermistor bolo-
meter detector covering a temperature range from -20° to +3,200°C.
Disks of Al_2O_3 are currently being prepared as standards for this
measurement method. The purities will range from 85 percent Al_2O_3
to 99 percent Al_2O_3. Once these disks are prepared and instrumented,
we will know the absolute accuracy of this method with respect to the
grid type thermocouple detectors.

Data reduction of dilatometer data on the suite of simulated lunar
rocks has started. There has been some delay, however, because of a
breakdown of an X-Y plotter early in the quarter, now repaired, and
requirements for other work during the quarter.

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R. Griffin remeasured dissipation factors for the simulated lunar rocks in an effort to check the spread of values determined previously. D. Lindroth rewrote the computer program for calculating the dissipation factor because of the Center's changeover to a new on-line computer. The program has been checked and works satisfactorily. Some of the measurements with the Q-meter did not seem to be consistent with previous measurements so the Q-meter was checked for accuracy of frequency setting. The instrument was found to be working properly so the remeasured data will be used with the computer program to calculate dissipation factor values. These values will be plotted against frequency to see if data spread has been reduced.

Status of Manuscripts

Dielectric Constants and Dissipation Factors for Fourteen Rock Types Between 20 and 100 Megahertz, by R. E. Griffin, is under preparation as a journal article.

Thermal Expansion Measurements of Simulated Lunar Rocks, by R. E. Griffin and D. P. Lindroth, is under preparation as a journal article.

STATUS REPORT FIRST QUARTER FISCAL YEAR 1969

Bureau of Mines NASA Program of Multidisciplinary Research

Task title: Basic problems of drilling in lunar environment
Investigator: Robert L. Schmidt, Project Leader
Location: Twin Cities Mining Research Center
                 Minneapolis, Minnesota
Date begun: January 1967   To be completed: December 1969

Personnel: William E. Bruce, Supervisory Mining Engineer
                 Robert L. Schmidt, Mining Engineer
                 Carl F. Anderson, Electronic Engineer
                 Robert R. Fumanti, Engineering Technician

PROGRESS REPORT

Objective

The objective is to study the basic problems of particle adhesion,
heat removal, and bit lubrication associated with drilling in a lunar
environment.

Progress During the First Quarter

A study is being made of the feasibility of performing a drilling test
in one of the ultrahigh vacuum chambers at the Twin Cities Mining Re-
search Center. The purpose of such an experiment is to determine if
particle adhesion in a vacuum exists to such an extent that mechanical
cuttings removal systems now contemplated for lunar drilling would be
ineffective. If feasible, the drilling experiment will be performed
first in atmosphere and then in ultrahigh vacuum with high-speed movies
and torque measurements taken in both environments.

During the first quarter, an experimental design was worked out, vacuum
equipment manufacturers were contacted regarding components of the ex-
perimental setup, and a layout drawing of the drilling apparatus was par-
tially completed.

James Paone, the original investigator for this task, has transferred
to the Bureau's Washington headquarters. William E. Bruce has assumed
supervision of the mechanical fragmentation research at the Center.
Mr. Schmidt has taken over the role of leading investigator for this
task.

Status of Manuscripts

None scheduled until the fourth quarter.

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STATUS REPORT FIRST QUARTER FISCAL YEAR 1969

Bureau of Mines NASA Program of Multidisciplinary Research

Task title: Use of explosives on the Moon
Investigator: Frank C. Gibson, Project Coordinator, Explosives Physics
Location: Explosives Research Center
                 Pittsburgh, Pennsylvania
Date begun: July 1966   To be completed: June 1969

Personnel: Frank C. Gibson, Supervisory Research Physicist
                 Richard W. Watson, Research Physicist
                 J. Edmund Hay, Research Physicist
                 Charles R. Summers, Research Physicist
                 William F. Donaldson, Research Physicist
                 John J. Mahoney, Laboratory Electrician
                 Elva M. Guastini, Explosives Equipment Operator

PROGRESS REPORT

Objective

The task objective is the acquisition of basic knowledge leading to the
solution of potential problems involved in the use of chemical high ex-
plosives in the lunar environment, specifically: (1) the stability of
explosives and explosive devices in the lunar environment; (2) the prob-
lem of exposure of explosives and explosive devices to (micro)meteoroid
impact; and (3) the propagation characteristics of the blast wave (prod-
ucts cloud) in the lunar atmosphere.

Progress During the First Quarter

During the previous quarter, the sensitivity of pressed granular ex-
plosive charges (Composition A-5, density 1.72 g/cm^3) to initiation by
projectile impact was determined for steel spheres having diameters from
0.5 to 0.0625 inches. The Slade-Dewey scaling law was found to best re-
late the mass and velocity of the particles for a 50 percent probability
of detonating the explosive. The equation, if reliable, could be used
to extrapolate data to meteorite velocities and provide an estimate of
the hazard from accidental initiation of explosives by meteorite impact.
However, the 0.0625-inch diameter spheres yielded velocities which ex-
ceeded the predicted value from the curve by 0.330 km/sec which may be
indicative of a critical size effect below which initiation by a single
particle may be very improbable. Hence, a modification of the existing
relationship is sought which will fit data in this range.

A projectile launcher has been designed to project 0.032-inch and smaller
diameter steel spheres but fabrication of the aluminum end plates to the

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proper thickness and angle and also the proper alignment of such small spheres in these liners is extremely critical. In many cases, the projectiles disintegrate before impacting the charges or in some instances they remain intact but miss the target. A precision lathe is currently being used to improve tolerance of the components.

Concurrent with the development of a small hypervelocity particle launcher, an apparatus has been fabricated for studying the properties of detonation products expanding into a vacuum. The vacuum system consists of a 260 liter/sec turbo-molecular pump connected by 6 feet of 2-inch diameter copper tubing passing through a bombproof wall to a vacuum chamber which encloses the charge to be tested. The test chamber includes a stainless steel cylinder, 4 inches inside diameter, 4 inches high, and 1/4-inch wall having a welded base plate on the bottom and an open top. The top is provided with a 6-inch square stainless steel plate with "O" ring grooves to accommodate either a 12.0 or 22.0 liter spherical glass flask. The top is bolted to the base plate and sealed to the stainless steel chamber with a 4-1/16-inch "O" ring. The periphery of the stainless steel cylinder is provided with both high and low voltage vacuum feed-throughs containing silver soldered Kovar seals. A quick-connect vacuum coupling, containing a Bayard-Alpert vacuum gage, is also welded to the vessel. The high voltage feed-throughs permit the explosive charge to be detonated by means of a 10 kv exploding wire initiator. The low voltage feed-throughs allow the pressure transducers, which are spaced at known distances from the charge periphery within the vacuum chamber, to be connected to their respective amplifiers and oscilloscopes for purposes of recording the velocities of the expanding products cloud. Supplementing this instrumentation and external to the vacuum chamber, a 15 kv exploding-wire back-lighting system is employed for photographing the expanding products cloud with either a 1 μsec Kerr-cell shutter camera or a high-speed framing camera.

Preliminary to the dynamic experiments, a 1-inch diameter by 1-inch long pellet of Composition A-5 (RDX + 1 percent wax) was subjected to vacuums of 5×10^{-7} to 5×10^{-8} torr over periods of as long as 72 hours. The results indicated that the vapor pressure of Composition A-5 was probably less than 10^{-8} torr at ambient temperature. The charge had a density of 1.6 g/cm^3 and was pressed from powder of which 100 percent passed through a #12 sieve and was retained on a #200 sieve, corresponding to a particle size distribution between 1680 and 74 microns. From such a particle size distribution it would be difficult to calculate the quantity of trapped air molecules which may have been removed from the interstices of the charge during evacuation.

Seven Composition A-5 charges with PETN centers have been detonated in air for the purpose of refining the instrumentation prior to vacuum firings. The charges were 1-inch diameter spheres of Compsoition A-5 with 1/4-inch diameter PETN cores containing the exploding wire initiators. Some difficulties have been encountered from stray electromagnetic

radiation from the back-lighting and initiating exploding wires and the Kerr cell. However, the most recent shots yielded fair agreement between product expansion velocities measured from Kerr-cell camera photographs and those obtained from the transducer records.

Status of Manuscripts

None in progress.

STATUS REPORT FIRST QUARTER FISCAL YEAR 1969

Bureau of Mines NASA Program of Multidisciplinary Research

Task title: Effect of lunar environment on behavior of fine particles
Investigator: David E. Nicholson, Project Leader
Location: Spokane Mining Research Laboratory
                Spokane, Washington
Date begun: April 1966   To be completed: June 1969

Personnel: David E. Nicholson, Mining Engineer
                Maynard O. Serbousek, Structural Engineer
                David F. Stafford, Physicist
                Robert W. Carnes, Engineering Technician
                Richard P. Curtin, Engineering Technician

PROGRESS REPORT

Objective

The primary objective is to determine basic physical properties which
may influence the handling and transportation of fine particles in a
lunar environment, as an extension of current studies of fine particle
behavior in mine backfill applications. Intergranular static and dy-
namic coefficient of friction and energy loss will be measured. Flow
rates and shear strength at various states of particle packing and at
various particle sizes will be determined and correlated with friction
and energy loss properties. This work will initially be performed un-
der conditions of normal earth atmosphere, but will be extended to in-
clude selected tests in ultrahigh vacuum. The work will be correlated
with the study of electrostatic properties of granular particles being
conducted at College Park and the study of frictional properties of
mineral surfaces being conducted at Minneapolis.

Progress During the First Quarter

Improvements in the milling circuit for the production of simulated
lunar rock powders were carried out during the first quarter. All the
crushing, grinding, screening, and classifying units were raised and
are accessible for repair work. New electrical conduits and an elec-
tronic tachometer installation will complete this conversion job.

Physical properties tests on the 14 simulated lunar rock powder sam-
ples prepared for the Jet Propulsion Laboratory, Pasadena, California
during the previous quarter were finished during the present quarter.

Work on the measurement of handling properties in moderate vacuum in-
cluded construction of a 10^{-5} torr glass flow container with cold trap.

Preliminary flow characteristics and packing and settling properties
were obtained on a simulated lunar basalt. These tests have not pro-
gressed sufficiently to report any results at this time but will be
detailed in the progress report to be assembled in the second quarter.

Status of Manuscripts

None in progress.

STATUS REPORT FIRST QUARTER FISCAL YEAR 1969

Bureau of Mines NASA Program of Multidisciplinary Research

Task title: Support for underground lunar shelter
Investigator: Lester J. Crow, Project Leader
Location: Spokane Mining Research Laboratory
                Spokane, Washington
Date begun: April 1966   To be completed: June 1969

Personnel: Robert C. Bates, Mining Engineer
                Lester J. Crow, Mining Engineer
                Edward W. Parsons, Mining Engineer
                Colen S. Smith, Mining Engineer

PROGRESS REPORT

Objective

The objective is to advance ground support technology in areas having
both terrestrial and extraterrestrial application. Research will be
conducted on ground support materials which have potential for utili-
zation both on the Earth and the Moon.

Progress During the First Quarter

The investigation of sulfur-basalt aggregates using combinations of
coarse and fine grain sizes has been completed. Manufactured samples
of basalt aggregate gradations, with selected average grain sizes and
coefficients of uniformity, did not produce compressive strengths ex-
ceeding those already obtained from natural grades of crushed basalt.
A series of experiments were made on medium sized particles (1/2 inch
to +4 mesh) combined with fine particles (-4 mesh) to obtain the best
gradation for a grout. The average compressive strength of a good
gradation of medium and fine particles was 9,400 psi.

Another series of experiments were made on sulfur with additives to
obtain expansion. The additives tried were in two groups, hydrocar-
bons and oxidizing agents. The oxidizing agents did not mix with the
molten sulfur (heated to 160°C) and did not impart expansion charac-
teristics to the solidified mass. Of the hydrocarbon group, R.C. 70
asphalt (30 percent Naptha) and sugar produced good results. When
the sulfur with the additive is heated to over 160°C hydrogen sulfide
gas starts to form. This process continues after the material is
poured into a mold. The net result is that the sulfur contains even-
ly distributed small gas pockets. On initial cooling open pockets
are formed on the top of the specimen. Then as the cooled material
reacts and expands over a period of hours these voids are filled and

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excess material is expelled and flows over the cooled surface. At the present time all of the particulars of this reaction are not known. Currently experiments are being conducted on a combination of aggregates and sulfur with additives.

Some experiments have been conducted with sodium silicate plus additives as a bonding agent, and by itself as a sealant on the surface of sulfur concrete cylinders, but it is too early in the experimental sequence to interpret results.

Preliminary analysis of other materials for use in ground support was carried out. Work on formulation of design concepts and devising techniques and equipment for fabrication or installation of support systems using sulfur and other materials was continued.

Mr. Crow has assumed duties as the leading investigator for this task.

Status of Manuscripts

Strengths of Sulfur-Basalt Concrete, by Lester J. Crow and Robert C. Bates, is being prepared as a Bureau Report of Investigations.

STATUS REPORT FIRST QUARTER FISCAL YEAR 1969

Bureau of Mines NASA Program of Multidisciplinary Research

Task title: Electrowinning of oxygen from silicate rocks
Investigator: Donald G. Kesterke, Acting Project Coordinator
Location: Reno Metallurgy Research Center
                 Reno, Nevada
Date begun: June 1966   To be completed: May 1969

Personnel: Donald G. Kesterke, Metallurgist
                 Freddy B. Holloway, Physical Science Technician
                 Carl H. Elges, Physical Science Aid

PROGRESS REPORT

Objective

The objective is to determine the feasibility of electrowinning ele-
mental oxygen from silicate rocks, as one phase of multidisciplinary
efforts to develop basic knowledge for using lunar mineral resources
in support of space missions.

Progress During the First Quarter

The objectives this quarter were to build and test a cell and insula-
tion system constructed of refractory materials which are inert to
elemental oxygen at elevated temperatures and to initiate experiments
to electrowin oxygen from silicates dissolved in a mixture of barium
and lithium fluorides.

Construction was completed on a new cell and insulation system. The
design incorporates a hot-pressed boron nitride crucible, with heat
furnished by silicon carbide resistors. Insulation surrounding the
cell consists of alumina, boron nitride powder, and conventional fire-
brick. Tests of the system showed it to be capable of temperatures
in excess of 1,400°C.

Experiments were initiated to prepare elemental oxygen by electroly-
sis of a fluoride-silicate mixture having a composition in weight-
percent of 69.4 BaF_2, 5.6 LiF, and 25.0 basalt plus sinter. Elec-
trolysis was conducted at about 1,150°C, with 15 to 28 amperes of
direct current applied to iridium electrodes. No oxygen was detected
above the cell, and severe corrosion of the cathode occurred. In a
subsequent experiment, a SiC cathode was used, and the iridium anode
surface area was doubled. Electrolysis was again conducted at 1,150°C,
with 25 to 47 amperes applied to the cell at an emf of 12 to 18 volts.

Analysis of the gases within the cell cover showed about 5 percent oxygen, while the atmosphere surrounding the cell insulation contained almost 3 percent oxygen.

Research to evaluate TiB_2 as an anode material showed it to be unsatisfactory. Complete corrosion of the electrode occurred after a short electrolysis, and the oxygen content over the cell was only about 1 percent.

Status of Manuscripts

None in progress.

STATUS REPORT FIRST QUARTER FISCAL YEAR 1969

Bureau of Mines NASA Program of Multidisciplinary Research

Task title: Reduction of silicates with carbon
Investigator: Larry A. Haas, Project Leader
Location: Twin Cities Metallurgy Research Center
                 Minneapolis, Minnesota
Date begun: June 1966   To be completed: May 1969

Personnel: Sanaa E. Khalafalla, Supervisory Research Chemist
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                 Howard W. Kilau, Chemist
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PROGRESS REPORT

Objective

The objective is to determine the optimum reaction rate criteria for
extracting oxygen from possible lunar materials in a simulated lunar
environment. The major emphasis of this research is to determine the
kinetics and mechanism of the carbothermal reduction of siliceous ma-
terials in a high temperature vacuum furnace.

Progress During the First Quarter

The objectives for this quarter were to complete the carbothermic
studies on alkali and alkaline-earth aluminum silicate minerals and
to determine the effect of temperature and carbon monoxide pressure
on the instantaneous carbothermic reduction rate of silica.

Previous studies conducted in the last quarter of fiscal year 1968
revealed that the carbothermal reducibility of complex aluminum sil-
icates increases with increasing silica content. When the mineral
contains appreciable amounts of alkali and alkaline-earth oxides,
the melting point of the silicate is lowered and the amount of reac-
tion becomes less dependent on the SiO_2 concentration, in addition
to being considerably diminished.

An attempt was made this quarter to investigate additional silicate
minerals and try to correlate the amount of reaction with the compo-
nent oxide concentration. A series of feldspars and feldspathoid
minerals were selected for these studies.

Tests were performed on sodium-calcium-aluminum silicates and the re-
sults are shown in the third block in table 1. The first two blocks
of this table give the results obtained in previous studies and are
introduced here for purposes of comparison. In this group of sili-
cates, the largest amount of CO was formed with the semisolid calci-
um feldspars-wollastonite and zeolite (5A). This is consistent with
the earlier finding that solid silicates reduce more easily than liq-
uid silicates. By contrast, very little reduction occurred with pure

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TABLE 1. - Carbothermic reduction data of silicate
minerals in 5 hours at 1,400°C

Mineral	Partial chemical analysis, percent						Milli-moles of CO formed	State of mineral at 1,400°C
	SiO_2	Al_2O_3	Na_2O	K_2O	CaO	MgO		
Si-O SYSTEM								
Wausau quartzite.	97.0	1.4	<0.1	<0.1	<0.1	<0.1	117	Solid
Do..............	97.0	1.4	<0.1	<0.1	<0.1	<0.1	102	Do.
Silica gel.......	98.7	<0.1	<0.1	<0.1	<0.1	<0.1	98	Do.
Fisher quartz....	99.1	<0.1	<0.1	<0.1	<0.1	<0.1	86	Do.
Si-Al-O SYSTEM								
Alumina (1139)[1]..	<0.1	96.3	<0.1	<0.1	0.7	<0.1	41	Solid
Alumina (38)[1]....	0.9	98.3	<0.1	<0.1	0.6	<0.1	46	Do.
Mullite..........	22.2	76.2	0.3	<0.1	<0.1	<0.1	64	Do.
Meta kaolin......	53.8	38.8	<0.1	<0.1	0.7	<0.3	69	Do.
Si-Al-Na-Ca-O SYSTEM								
Lime.............	1.4	0.4	<0.1	<0.1	99.6	0.1	4	Solid
Gehlenite........	34.8	14.0	0.1	<0.1	33.5	5.4	54	Semisolid
Zeolite (5A)[1]....	43.4	39.1	4.1	0.4	10.1	-	75	Do.
Zeolite (4A)[1]....	44.1	41.4	14.7	0.3	0.9	0.1	19	Liquid
Wollastonite.....	50.7	3.6	<0.1	<0.1	45.2	0.2	91	Semisolid
Bytownite........	51.6	18.1	<0.1	0.3	6.9	4.2	7	Liquid
Labradorite......	57.1	27.0	3.8	0.7	0.3	0.1	8	Do.
Oligoclase.......	63.2	21.8	7.0	0.8	4.5	0.1	5	Do.
Albite...........	65.7	15.6	10.0	0.3	1.8	0.2	15	Do.
Si-Al-Na-K-Ca-O SYSTEM								
Mica.............	46.9	44.3	0.9	9.1	1.1	0.6	98	Semisolid
Orthoclase.......	61.8	17.4	0.2	11.3	4.1	0.2	4	Liquid
Feldspar (1775)[1].	66.7	17.1	4.9	4.2	2.2	-	9	Do.
Obsidian.........	70.7	10.2	3.4	3.2	2.0	0.3	8	Do.
Si-Al-Na-Ca-Mg-O SYSTEM								
Magnesia.........	0.6	0.2	1.0	0.2	1.2	96.7	2	Solid
Hornblende.......	21.6	64.8	1.0	<0.1	4.5	6.7	1	Liquid
Olivine..........	40.5	0.7	0.2	<0.1	0.2	45.6	20	Do.
Pyroxene.........	52.0	1.7	1.0	0.2	20.4	15.3	0	Do.
Mg trisilicate...	64.8	0.8	2.8	0.2	2.4	25.9	111	Semisolid
Talcum...........	65.1	1.1	<0.1	<0.1	1.5	29.8	135	Do.

[1]Identification symbols for different minerals.

lime even though it retains its solid state of aggregation at the re-
action temperature. The unusually high melting point of CaO and the
extreme stability of the calcium-oxygen bond, as typified by the large
negative enthalpy and free energy of formation, accounts for the sta-
bility of lime toward carbothermal reduction as well as to the retain-
ing of solid or semisolid state of aggregation for minerals rich in
that component. The inverse correlation between oxide reducibility
and melting point was reported by Elyutin.[1] To help in assessing the
effect of oxide constituents on the gross properties of complex sili-
cates, thermodynamic data as well as melting points of some pertinent
oxides are compiled in table 2.

TABLE 2. Thermodynamic data and melting
points of oxides

Oxide	$-\Delta F_f^{o}$ [1]	$-\Delta H_f^{o}$ [1]	Melting point,[2] $^{\circ}C$
K_2O	2	241	[3]350
Na_2O	50	60	[3]1,275
Fe_2O_3	64	130	1,565
Fe_3O_4	70	130	[3]1,538
FeO	74	110	1,420
SiO_2	139	207	1,700
Al_2O_3	181	267	2,015
MgO	184	350	2,800
CaO	219	305	2,580

[1]Standard enthalpy and free energy changes
  in kcal per gram-mole of oxygen at
  1,400°C taken from reference 2.
[2]Values taken from reference 3.
[3]Oxides decompose at these temperatures.

The sodium feldspars: zeolite 4A, labradorite, oligoclase, and albite
contain very little lime and melt easily. Their extent of reduction
appears to exhibit a systematic dependence on their Na_2O content. The
bond energy and melting point of Na_2O, being consistently lower than
those of silica and lime, tends to make this component the determining
factor for total reducibility. Millimoles of CO formed in 5 hours from
zeolite 4A, albite, oligoclase and labradorite are 19, 15, 5, and 8,
respectively; their Na_2O content are seratim: 14.7, 10.0, 7.0, and 3.8
percent. Although oligoclase should reduce more readily than labrador-
ite, its higher lime content of 4.5 versus 0.3 percent for labradorite
will reverse the trend.

[1]Elyutin, V. P., P. F. Markulova, and Yu. A. Pavlov. (Temperature of
  the Beginning of Metal Oxide Reduction by Solid Carbon.) Proizvodstve
  i Obrabotka Stali Splavov, Metallurgizat, Moscow, v. 38, 1958, pp. 79-87.
[2]Elliott, J. F. and M. Gleiser. Thermochemistry for Steelmaking. Addi-
  son Publishing Co., Inc., London, v. 1, 1960, 296 pp.
[3]Handbook of Chemistry and Physics. Ed. by R. C. Weast. Chemical Rubber
  Co., 47th edition, 1966-67, pp. B150-255.

Some potash feldspars were also investigated and the results are given in the fourth block of table 1. Only mica retained its solid state at 1,400°C and hence reduced more readily than orthoclase, feldspar (1775) and obsidian which melted. Although potassium oxide has the lowest melting point, yet its enthalpy of formation is more negative than that of silica, and hence no conclusive pattern can be found for the dependence of silicate reducibility on its potassium oxide content.

Results with magnesium calcium silicates are given in the fifth block of table 1. Here only talcum and magnesium trisilicate remained solid at 1,400°C and hence their reducibility rose well above the others that melted. Although magnesia remained solid, yet its reducibility was very poor due to its exceptionally high melting point and low free energy and enthalpy of formation.

The foregoing comparisons were obtained on similarly constituted silicates, i.e., in the same block in table 1. Cross examination among the various blocks revealed additional information. Comparison of the second and third blocks shows that although alumina reduced faster than lime, yet aluminum silicate (meta kaolin) reduced slower than calcium silicate (wollastonite). This is undoubtedly related to the melting point of calcium silicate of about 1,500°C, being lower than that of aluminum silicate of about 1,800°C. Thus, in addition to the melting point of the constituent oxides, the melting point of the mineral silicate appears to exert its effect on carbothermal reducibility.

When the silicate reducibility was plotted against the percent of silica in the mineral, the curves of figure 1 were obtained. Curve (a) groups together those silicates which retain their solid or semisolid state of aggregation at 1,400°C, while curve (b) relates to minerals which melt at this temperature. Although reducibility generally increases with increasing silica content, yet the relation is confused by the side effects of lime and magnesia constituents. Straight-line relationships were obtained during the last quarter only with aluminum silicates, devoid of lime and magnesia. It appears that the additivity rule for the reducibility of complex silicates cannot be deduced from simple two-dimensional diagrams, but requires multi-dimensional surfaces, with each axis describing the concentration of an individual oxide, barring some complications that could arise from unforeseen deviations in the melting points of the minerals.

During this quarter, instantaneous kinetic curves for the reaction of silica with graphite were recorded by the newly acquired fast-heating vacuum furnace and a quartz spring balance. The effect of temperature on the reaction rate was first investigated. The plot of percent weight loss, W, versus time at four temperatures from 1,375° to 1,450°C and under a furnace pressure of about 10^{-6} torr, gave the kinetic curves of figure 2. It appears that the reaction starts with an induction period which gets progressively longer the lower the reduction temperature
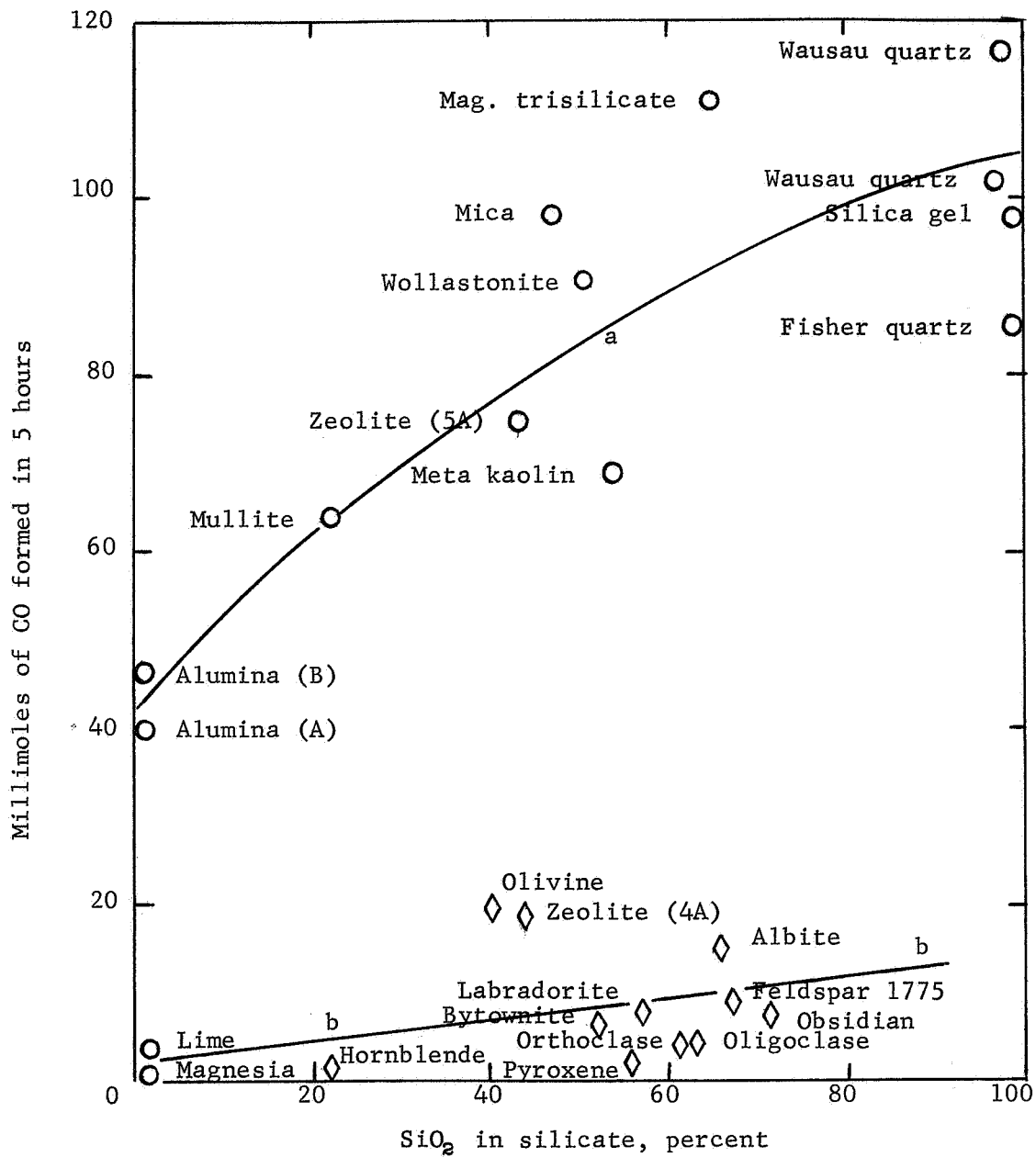
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FIGURE 1. - Reducibility at 1,400°C of Silicates
as a Function of Their Silica Content.

a - Silicates that retain their solid
or semisolid state.
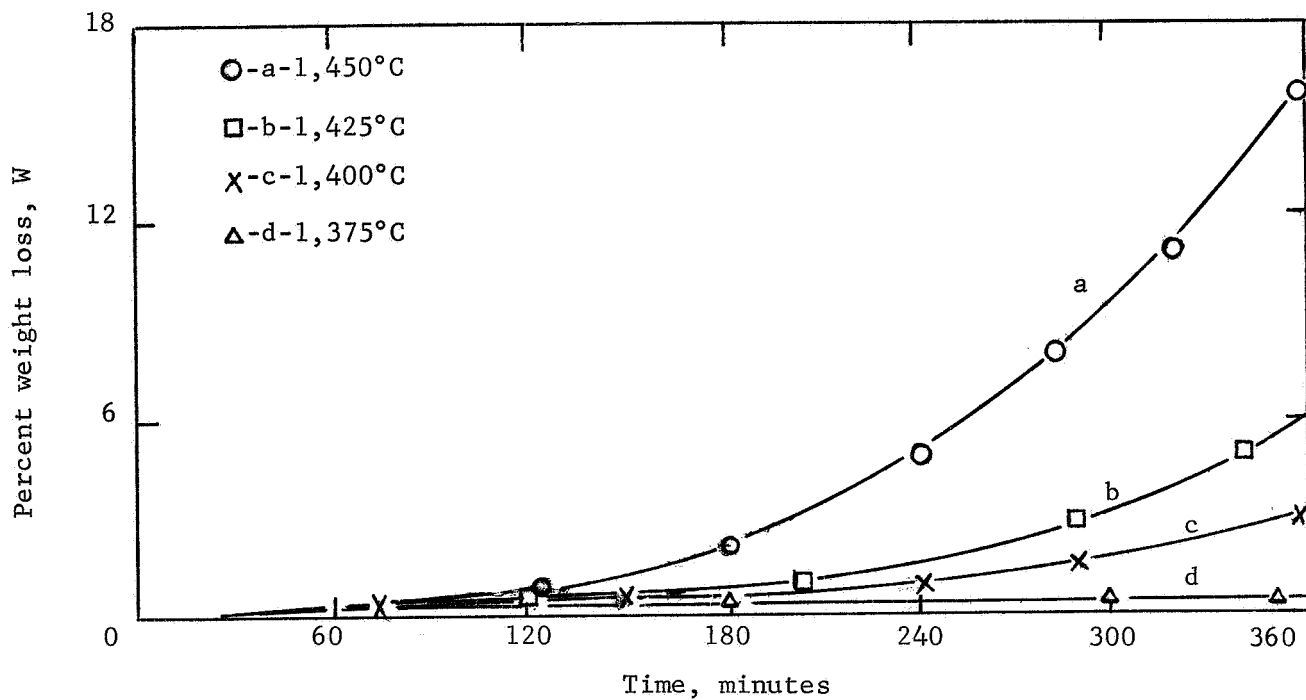b - Silicates that are melted at the
reduction temperature.

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FIGURE 2. - Kinetic Curves for the Reaction of Silica with
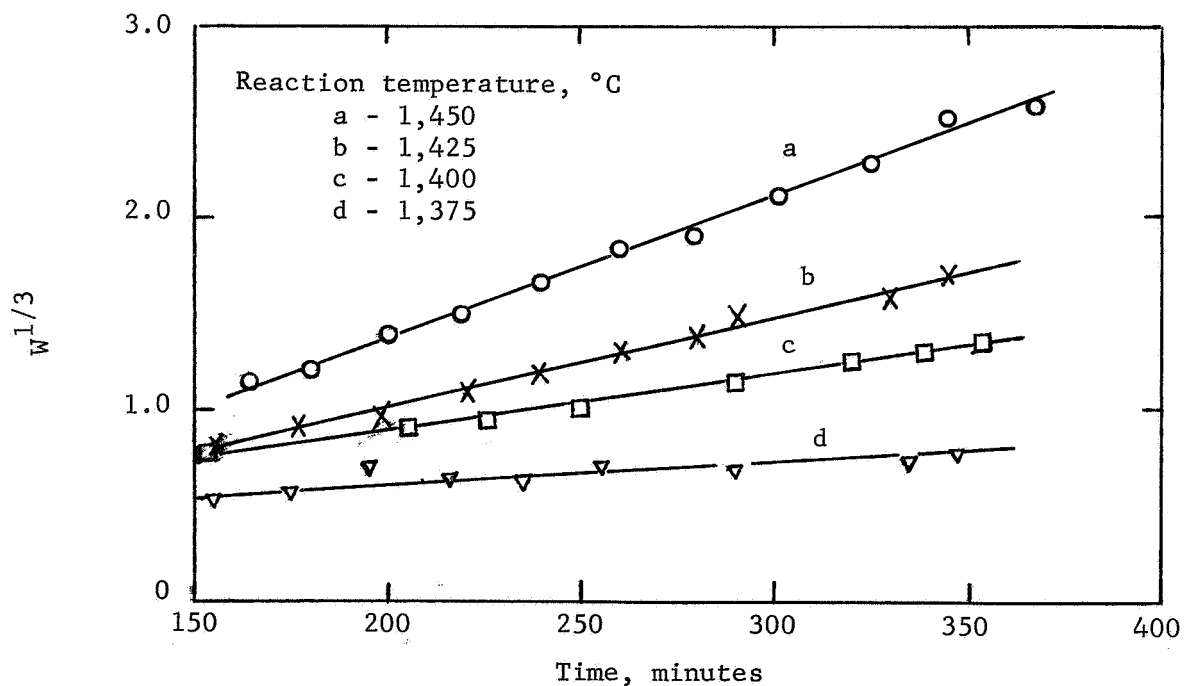Graphite at Various Temperatures.

FIGURE 3. - Variation of Cubic Root of Percent
Weight Loss with Time.

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and which amounts to about 2 hours at 1,450°C. This is followed by an acceleratory period. Analysis of the curves in figure 2 showed that the parabolic rate-law did not apply since W^2 was not proportional to the reaction time; and, hence, the reaction cannot be diffusion controlled (solid state diffusion). It was found that the plot of $W^{1/3}$ against time at the post-induction period gave reasonably good straight lines as shown in figure 3. After the slow induction period, the reduction curve seems to follow the equation

$$W^{1/3} = a + bt, \tag{1}$$

where a and b are constants. The rate of carbothermal reduction would then be given by

$$\Re = \frac{dW}{dt} = 3b \; W^{2/3} = k \; W^{2/3}. \tag{2}$$

The most general equation describing solid-state reactions is given by

$$\frac{dW}{dt} = k \; W^\alpha \; (1-W)^\beta \;, \tag{3}$$

with α and β constants, called the homogeneity factors and W now stands for the fraction of the initial compounds reacted. It appears that for the carbothermal reduction of silica $\beta = 0$ and $\alpha = 2/3$. Prodan and Pavlyuchenko[4] reported values of β obtained by different authors to be 0, 1/3, 1/2, 2/3, 1, and 2, while α had values of 0, 2/3, and 1. Although this does not throw much light on the reaction mechanism, yet it served to linearize the post-induction reduction curve. The slopes of the lines in figure 3 give a direct measure of the isothermal reaction rate constant. An Arrhenius plot of the slopes is shown in figure 4 and indicates an activation energy of 123 kcal. This agrees well with the value of 122 kcal reported by Klinger.[5]

To study the effect of CO pressure on the reaction rate, small amounts of this gas were leaked in the furnace chamber at the rate required to maintain a finite steady pressure in the chamber. Tests were conducted at 1,400°C and with carbon monoxide pressures of 0.25, 0.02, 10^{-3} and 10^{-6} torr. The reduction-time curves are shown in figure 5. The blank test given by curve (a) represents the sum of two separate runs for silica alone and graphite alone with no added carbon monoxide in the chamber. The amount of reaction with the blank was about the same as that obtained with a silica-graphite mixture at a CO pressure of 0.25 torr curve (b), even though the equilibrium pressure for this reaction is 76 torr. Therefore, the furnace pressure must be about three orders of magnitude less than the equilibrium pressure for a measurable reaction to occur. The rate of reaction at 5 hours was about 20 times faster at 10^{-6} torr than at 0.25 torr, indicating a significant effect of pressure on the reaction rate.

[4] Prodan, E. A. and M. M. Pavlyuchenko. (Heterogeneous Chemical Reactions.) Nauka i takhnika, Minck, USSR, 1965, p. 20.

[5] Klinger, N., E. L. Strauss and K. L. Komarek. Reactions Between Silica and Graphite. J. Am. Chem. Ceram. Soc., v. 49, No. 7, 1966, pp. 369-375.
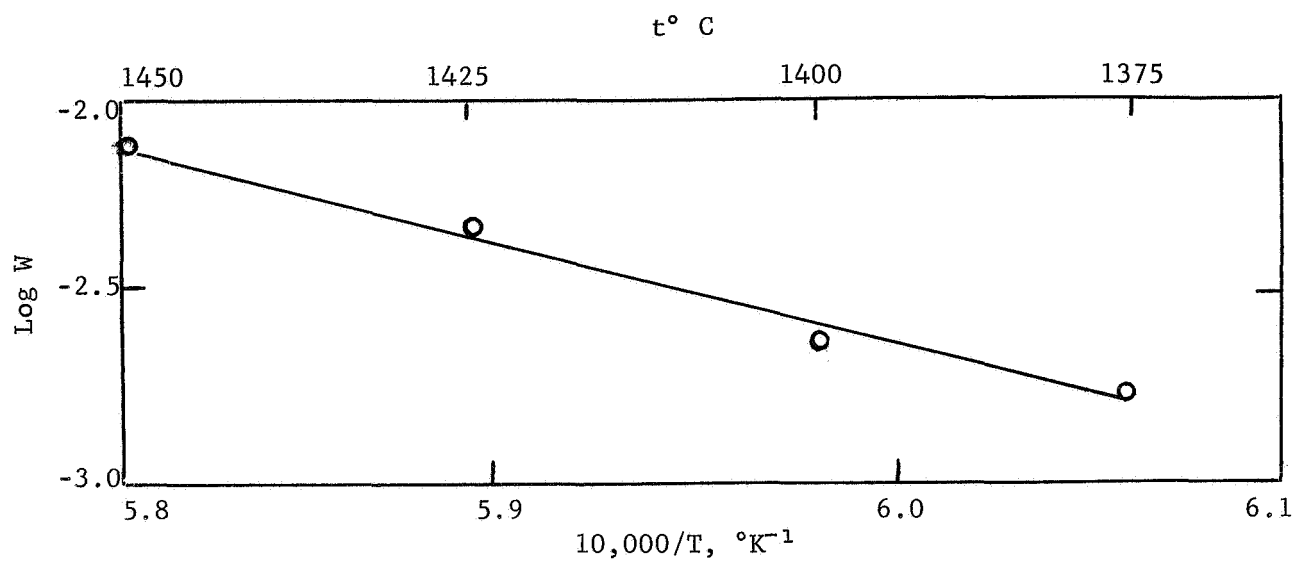
t° C

FIGURE 4. - Arrhenius Plot of the Carbothermic Reduction Data.

Pressure of carbon monoxide:
a - blank, b - 0.25 torr, c - 0.02 torr.
d - 10^{-3} torr and
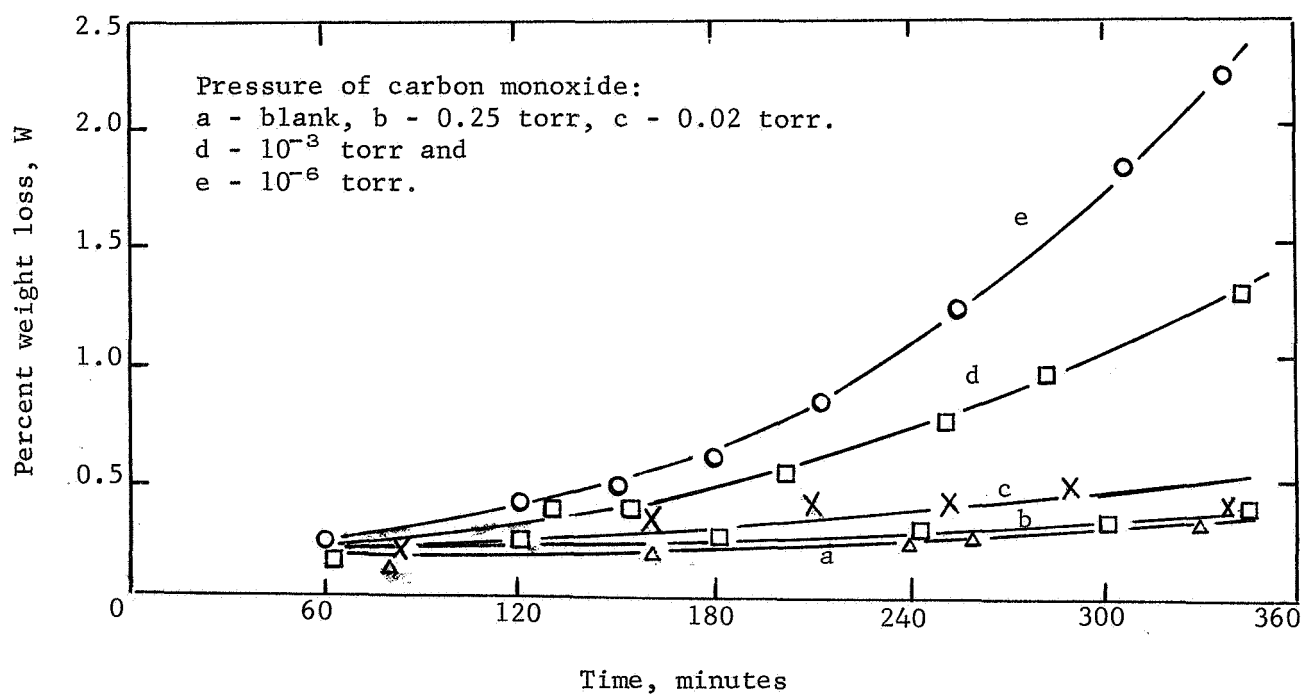e - 10^{-6} torr.

Time, minutes

FIGURE 5. - Effect of Carbon Monoxide Pressure
on the Graphite Silica Reaction.

The gaseous reaction products were analyzed with an Ultek quadrapole residual gas analyzer. The gases liberated from the individual reactants were first examined. Small amounts of CO, atomic mass units (amu) = 28, were obtained with graphite, apparently due to its reaction with residual air in the furnace. With the silica blank, small amounts of Si, amu = 28, and SiO, amu = 44, were observed. When the silica-graphite mixture was used, one large peak appeared at the position corresponding to an atomic mass unit of 28 which corresponds to singly ionized carbon monoxide and/or monoatomic silicon vapor. No recognizable peak from the ambient noise was observed at position 44, indicating the absence of carbon dioxide and silicon monoxide in the reaction products. Hence, the principle gaseous products of this reaction are carbon monoxide and silicon.

Mr. Haas has taken over from Dr. Khalafalla the role of leading investigator.

Status of Manuscripts

The Effect of Physical Parameters on the Reaction of Graphite with Silica in Vacuum, by L. A. Haas and S. E. Khalafalla, is near to publication as a Bureau Report of Investigations.

STATUS REPORT FIRST QUARTER FISCAL YEAR 1969

Bureau of Mines NASA Program of Multidisciplinary Research

Task Title: Stability of hydrous silicates and oxides in lunar
               environment
Investigator: Hal J. Kelly, Project Coordinator
Location: Albany Metallurgy Research Center
               Albany, Oregon
Date begun: April 1966   To be completed: May 1969

Personnel: Hal J. Kelly, Supervisory Ceramic Research Engineer
               Ray L. Carpenter, Research Physicist

PROGRESS REPORT

Objective

The objective of this project is to investigate by differential ther-
mal analysis (DTA) and thermogravimetric analysis (TGA) the stability
of some silicate and oxide minerals in air and under high vacuum at
elevated temperatures. The determination of the energies required
to dissociate oxide and silicate minerals and recover oxygen and water
is the long range objective.

Progress During the First Quarter

The objectives for the quarter were to install and calibrate the plat-
inum calorimeter, to improve the operation of the DTA equipment, and
to determine the heat of reaction of prefired kaolinite and the heat
of decomposition of epidote in air.

A reduction in noise on the DTA curve was made by installation of a
single water pipe ground with all components hooked to it and by chang-
ing the furnace input circuit to eliminate a ground loop circuit. Fur-
ther reduction in noise level was obtained by adjustment of the pro-
portioned band width. This also improved the heating rate at low tem-
peratures. The base line drift was checked in air and vacuum and
found to be 25 microvolts or less in air and 50 microvolts or less in
vacuum.

Comparison of response of 1/2- and 3/4-inch tall platinum crucibles
was measured and the 1/2-inch high crucibles were found to give the
larger response per gram of sample. Calibration of the platinum cal-
orimeter was made with crucibles. A calibration factor of 1.12 calo-
ries per unit area was obtained.

The heat of reaction of prefired kaolinite was found to be 31.3 calories per gram, and the heat of decomposition of epidote in air was found to be 72.7 calories per gram.

Status of Manuscripts

None scheduled until the third quarter.

STATUS REPORT FIRST QUARTER FISCAL YEAR 1969

Bureau of Mines NASA Program of Multidisciplinary Research

Task Title: Magnetic and electrostatic properties of minerals in
                a vacuum
Investigator: Foster Fraas, Project Leader
Location: College Park Metallurgy Research Center
                College Park, Maryland
Date begun: June 1966   To be completed: May 1969

Personnel: Ronald A. Munson, Research Chemist
                Foster Fraas, Metallurgist
                Anderson Walls, Laboratory Technician

PROGRESS REPORT

Objective

The objective is to study adsorption and contact electrification in a
vacuum and determine their effect on the separability of nonconducting
minerals.

Progress During the First Quarter

The objectives for this quarter were to measure flow characteristics
under ultrahigh vacuum with particles of quartz at a size smaller than
previously used and to initiate preparation of a manuscript.

The program was delayed by two factors, the burn-out of the vibration
drive unit coil which had to be returned to the manufacturer for re-
pair, and the interruption of the overnight operation of the pump by
power failures resulting from summer storms. The turbomolecular pump-
ing system includes automatic cut-out relays and vacuum chamber back-
fill valves for protection against a sustained power failure during
which there is danger of the forepump oil rising into the turbomolec-
ular pump.

The previously reported results on minerals in a size range of minus
35 plus 65 mesh were repeated with quartz in the size range of minus
65 plus 150 mesh. No significant adhesion to transport surfaces was
noted at the ultrahigh vacuum of 7.5×10^{-8} torr. Some single parti-
cle layer adhesion which did occur near the bottom of the feed hopper
corresponded to the behavior usually obtained in the low humidity at-
mospheres.

Quartz is the most negative of the simulated lunar mineral group which includes microcline, olivine, and hornblende. It is most likely to adhere since it also has the largest magnitude of charge. A vibrating feeder with a contacting surface more negative in contact potential value than the aluminum surface used in these tests would decrease the magnitude of the quartz charge and may change microcline to a positive electrification polarity.

Contact electrification under vacuum, summarized in table 1, paralleled the results with coarser particle sizes. The average particle charge is also of the same order of magnitude when based on the same density of surface charge. Contact electrification is reversible after return to one atmosphere and is much less than for the coarser size particles. This is attributed to the difference in atmospheric humidity. It is probable that the low humidity of 29 percent in the coarser size tests delayed adsorption of atmospheric moisture and provided a contact electrification similar to that for a vacuum.

Status of Manuscripts

Mineral Separation in a Vacuum, by F. Fraas, is under preparation as a journal article.

TABLE 1. - Contact electrification of quartz in vacuum and in air

Mineral	Initial atmospheric pass[1] Potential (volts)	First pass under vacuum[2]			Second pass under vacuum[3]		Pass after sample returned to atmosphere[4]		
		Pressure (10^-7 torr)	Potential (volts)	Average particle charge coulombs (10^-14)	Pressure (10^-8 torr)	Potential (volts)	Weight (grams)	Potential (volts)	Potential[5] (volts)
Quartz	-14.0	4.0	-42	1.8	7.5	-36	1.867	-12.0	-12.9

[1] Initial weight of sample = 2.000 grams. Size range is minus 65 plus 150 mesh. Initial conditions 56 percent relative humidity at 29°C.

[2] First pass conducted with sample at room temperature and no intervening increase in temperature.

[3] Second pass conducted at room temperature but with an intervening 18 hour sample temperature of 102±2°C.

[4] Final conditions 52 percent relative humidity at 30°C.

[5] Calculated for 2 grams.